Fabrication and characterization of SiO$_2$ microcantilever for microsensor application

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Abstract

SiO$_2$ microcantilevers were developed to improve the sensitivity of microcantilever sensors. Silicon plasma dry etching technique was employed to release SiO$_2$ cantilever from bulk silicon at high rate using an ordinary 2 μm thickness of SiO$_2$ covered silicon wafer. The V-shaped microcantilever is 200 μm long, 25 μm wide, and 2 μm thick. The spring constant of the cantilevers was measured to be 0.104 N/m. Significant deflection amplitude of the microcantilever was observed upon exposure of low concentration of aminoethanethiol due to its low spring constant.

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1. Introduction

Microcantilevers have recently attracted considerable interests in the development of a wide range of novel physical [1], chemical [2], and biological [3] sensors. Microcantilever deflection is one unique transduction mechanism of microcantilever sensors. The technology is based upon changes in the deflection property induced by environmental factors in the medium in which a microcantilever is immersed. It has been observed that microcantilevers bend upon the specific binding of species in the environment due to an adsorption induced surface stress change [4]. This concept has already been used to demonstrate the feasibility of chemical detection of a number of vapor phase analytes as well as highly sensitive detection of chemical and biological species in solutions.

Surface modification by self-assembled monolayer (SAM) has proven to be a successful method for surface modification of microcantilever for sensing applications [5,6]. In these microcantilever sensors, molecular recognition agents containing thiol compounds or silanes have been synthesized for the preparation of SAM on the microcantilever. Nanometer scale microcantilever bending was observed in most of SAM modified microcantilever sensors developed recently [3,6]. A 200 μm-long silicon microcantilever modified with self-assembled monolayers generally bends 10–200 nm upon binding of analytes in solutions with monolayer receptors on the microcantilever surface. At such nano-scale bending, especially for 10 nm bending amplitude, noise (1–5 nm in general) is a serious problem for accurate concentration measurement.

The weak microcantilever bending amplitudes are due to both weak surface stress change and large spring constant of the microcantilever. First, most of the adsorption induced forces on the SAM modified microcantilevers were not significant enough to produce a large microcantilever bending amplitude. Specific molecular bindings on the SAM modified microcantilevers normally produce surface stress at nano Newton (nN) levels. Recently, surface modification chemistries that could generate significant surface stress changes are under development in order to improve the cantilever bending amplitude [7]. Second, most microcantilever chemical and biological sensors developed recently were based on silicon microcantilevers. Silicon material has a relative large spring constant that resists microcantilever bending, especially when the surface stress change is rather small. It is anticipated that a microcantilever made of a more elastic material with a smaller spring constant, such as silicon dioxide, could give larger bending amplitude under the same condition as silicon cantilevers. The Young’s modulus of silicon dioxide is 76.5–97.2 GPa for SiO$_2$, which is much smaller than that of silicon at 155.8 GPa [8]. In this work, we report the fabrication and characterization of silicon dioxide microcantilevers. Although other elastic materials...
2. Experiments

2.1. Materials

Commercially available silicon microcantilevers were purchased from Veeco Instruments, CA, USA. The dimensions of the V-shaped silicon microcantilevers were 180 μm length, 25 μm width, and 1 μm thickness. One side of the cantilever had a thin film of chromium (3 nm) followed by a 20 nm layer of gold deposited by e-beam evaporation. Another side of the microcantilever was made of silicon with a thin, naturally grown oxide layer. Aminoethanethiol hydrochloride was used as received from Aldrich. High-purity de-ionized water was obtained with a Milli-Q water system (Millipore).

2.2. Fabrication procedure

The dimensions of the designed V-shaped SiO₂ microcantilevers were 200 μm in length, 25 μm in width, and 2 μm in thickness. The width at the root was 150 μm. A round tip, which has 25 μm radius, was specifically designed at the tip of cantilever to reflect the incident laser. In order to lower the noise level, 2 μm thickness was designed to give better mechanical strength.

The fabrication process has two steps: first, pattern the cantilever beam on SiO₂ layer by photolithography and buffered oxide etching (BOE); second, release the cantilever from bulk silicon by dry plasma etching. The fabrication process of SiO₂ cantilever is depicted in Fig. 1. Shipley 1813 positive tone photoresist was spun on the surface of a 500 μm thick silicon wafer with 2 μm thick SiO₂ layer (Fig. 1a). A microcantilever beam pattern was transferred to the photoresist layer on the front side of wafer by standard photolithography process and then the SiO₂ cantilever beams were formed by etching with buffered oxidation etchant (BOE HF:HNO₃ = 1:6). In the mean time, the entire SiO₂ layer on back side was etched off (Fig. 1b). The photoresist was then cleaned by acetone and DI water (Fig. 1c). A 20 μm thickness of photoresist AZ9260 was spun on the backside of wafer and then the back side wafer was patterned with photolithography process (Fig. 1d). The thick photoresist pattern served as a mask for deep silicon plasma etching. The wafer was etched off by inductive coupling plasma (ICP) process to release microcantilever beams from bulk silicon (Fig. 1e). In our system, the plasma source was generated by an inductively coupled plasma source manufactured by Alcatel Comptech Inc.

2.3. Deflection measurement

The deflection experiments were performed in a flow-through glass cell (Digital Instruments, CA, USA), such as those used in atomic force microscopy. The microcantilever was immersed in distilled water. Initially, water was circulated through the cell using a syringe pump. A schematic
diagram of the apparatus used in this study was reported before [5]. A constant flow rate of 4 ml/h was maintained during the entire experiment. Aminothanethiol solution was injected directly into the fluid flow via a low pressure injection port/sample loop arrangement. This arrangement allowed for continuous exposure of the cantilever to the desired solution without disturbing the flow cell or the flow rate. The deflection measurements were carried out with an AFM photodiode. The bending of the cantilever was measured by monitoring the position of a laser beam reflected from the cantilever onto a four-quadrant photodiode. The cantilever was immersed in water until a stable baseline was obtained and the voltage of the position sensitive detector was set as background corresponding to 0 nm.

3. Results and discussion

3.1. Fabrication

Wet etching was initially used in our experiment to fabricate SiO2 microcantilevers. The fabrication process was unsuccessful. Due to the low selectivity of low SiO2:Si etching selectivity using either KOH or EDP etchants, the processing conditions were hard to control in order to selectively etch off 500 \( \mu \)m thickness of silicon while keeping 2 \( \mu \)m thickness of SiO2 unaffected.

Dry plasma etching offers many advantages over wet chemical etching methods, including high etch rate, compatibility with traditional IC processing [10], high Si:SiO2 etching selectivity [11] (up to 350:1). The high selectivity is a distinct advantage in releasing SiO2 cantilever beams from bulk silicon. Deep dry plasma etching achieves the high etching rate and straight sidewall. This process simplifies the device design and fabrication processing. The plasma had high energy in the vertical direction, so the passivation layer (photoresist) was removed from the horizontal surface much faster than the vertical direction. After the passivation layer (photoresist) was removed from the horizontal surface, the bulk silicon was exposed and etched by the high-energy plasma and SiO2 microcantilever beams were successfully released from bulk silicon (Fig. 2).

3.2. Characterization

The spring constant of the SiO2 cantilevers was measured to estimate their performance in chemical sensing. Methods for microcantilever spring constant measurement have been well illustrated before [12-15]. Using a cantilever of known spring constant as reference was also commonly used [14,15]. In our work, a commercially available calibrated silicon cantilever [10] was used as a reference cantilever and its spring constant was known to be 0.26 N/m. The silicon cantilever was horizontally placed on a fixed substrate. Fabricated SiO2 cantilever was horizontally placed on the same substrate while having a different altitude level from that of silicon cantilever. The SiO2 cantilever beam was placed in contact with the silicon cantilever beam (Fig. 3). Both cantilevers deflected due to the contact force against each other. The forces applied on the two cantilever beams were the same while their directions were opposite, and the cantilever deflection was inversely proportional to its spring constant,

\[
y = \left( \frac{1}{k} \right) F
\]

where \( y \) is the deflection of the cantilever at the end and \( F \) is the force applied at the end. As shown in Fig. 3, the deflection of silicon cantilever tip was about 40 \( \mu \)m and the deflection of SiO2 cantilever was 101 \( \mu \)m. Therefore,
The spring constant of SiO₂ cantilever was calculated to be
\[ \frac{40 \mu m}{101 \mu m} \times 0.26 \text{N/m} = 0.104 \text{N/m} \]

The shape and mechanical properties of both cantilevers are shown in Fig. 4 and Table 1, respectively.

The resonance frequency of the microcantilever beam was measured to be 22.5 kHz.

The resonance frequency of both SiO₂ and silicon microcantilever are also compared in Table 1.

Although the thickness of the SiO₂ microcantilever was double that of silicon microcantilever, the spring constant of the SiO₂ cantilevers was 2/5 of that of the commercially available silicon cantilevers, suggesting that a SiO₂ cantilever has the potential to give stronger bending amplitude than silicon cantilever as chemical or biological sensors. Microcantilever bendings of silicon and SiO₂ microcantilevers to the same concentration of aminoethanethiol solution was used to compare the bending amplitudes of the silicon and SiO₂ cantilevers. Both cantilevers were coated with a thin layer of gold (20 nm) on one side. It was known that thiol compounds, such as aminoethanethiol, form a monolayer on the gold surface that bends the microcantilever [22].

As shown in Fig. 5, the deflection of silicon cantilever upon exposure to 10⁻⁵ M of aminoethanethiol solution was approximately 90 nm, while that for SiO₂ cantilever was 510 nm. The 2 μm SiO₂ microcantilever bent 5.6 times of that of 1 μm silicon microcantilever upon exposure to the same concentration of an aminoethanethiol solution. Since the two cantilevers have very close geometry shapes and dimensions, it is expected that the surface stresses induced on the two cantilevers are the same (σSiO₂ = σSi). Thus

\[ \frac{\Delta Z_{\text{SiO₂}}}{\Delta Z_{\text{Si}}} = 1.15 \times \frac{L_{\text{SiO₂}}}{L_{\text{Si}}} \times \frac{t_{\text{SiO₂}}}{t_{\text{Si}}} = 5.18 \]

where \( \Delta Z \) is the observed deflection at the end of the cantilever, \( v \) is Poisson’s ratio, \( t \) is the thickness of the cantilever, \( L \) is the length of the cantilever, and \( E \) is the differential stress on the cantilever.

And, the spring constant for a cantilever is

\[ \kappa = \frac{Ew^3}{4L^3} \]

where \( w \) is width of a cantilever, and \( \kappa \) is spring constant of a cantilever. Eq. (2) is then derived as

\[ \Delta Z = \left( \frac{1}{(Ew^3/4L^3)} \right) \left( \frac{3(1 - \nu)E}{4} \right) t \left( \frac{3(1 - \nu)w}{4L} \right) s \]

The average \( (1 - \nu) \text{SiO₂}/(1 - \nu) \text{Si} \) is 1.15 [18–21]. Since the two cantilevers have very close geometry shapes and dimensions, it is expected that the surface stresses induced on the two cantilevers are the same (σSiO₂ = σSi). Thus

\[ \frac{\Delta Z_{\text{SiO₂}}}{\Delta Z_{\text{Si}}} = 1.15 \times \frac{L_{\text{SiO₂}}}{L_{\text{Si}}} \times \frac{t_{\text{SiO₂}}}{t_{\text{Si}}} = 5.18 \]

The bending responses of the silicon and SiO₂ microcantilevers upon exposure to a 10⁻⁵ M solution of aminoethanethiol in ethanol.

Table 1

<table>
<thead>
<tr>
<th>Cantilever type</th>
<th>Length (μm)</th>
<th>Width (μm)</th>
<th>Thickness (μm)</th>
<th>Spring constant (N/m)</th>
<th>Resonance frequency (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂ cantilever</td>
<td>200</td>
<td>25</td>
<td>2</td>
<td>0.104</td>
<td>22.5</td>
</tr>
<tr>
<td>Commercial silicon cantilever</td>
<td>180</td>
<td>25</td>
<td>1</td>
<td>0.26</td>
<td>40</td>
</tr>
</tbody>
</table>
This analysis fits well with our observation of microcantilever bending amplitude upon exposure to $1 \times 10^{-9}$ M aminoethanethiol solution.

4. Conclusion

In this paper, we report the fabrication process and characterization of SiO$_2$ microcantilevers. Silicon plasma dry etching was used to release SiO$_2$ cantilever beam from bulk silicon. The SiO$_2$ cantilevers fabricated have lower spring constant compared to commercially available silicon microcantilevers. The fabricated SiO$_2$ cantilevers could be used for the development of macrocantilever chemical and biological sensors with much higher sensitivity than standard silicon microcantilevers.

References
